Status of the UV Cure Powder Coating Demonstration Project







maintaining the data needed, and c including suggestions for reducing	lection of information is estimated to ompleting and reviewing the collecti this burden, to Washington Headqu uld be aware that notwithstanding an DMB control number.	ion of information. Send comments is arters Services, Directorate for Infor	regarding this burden estimate of mation Operations and Reports	or any other aspect of the 1215 Jefferson Davis	is collection of information, Highway, Suite 1204, Arlington			
1. REPORT DATE 2012		2. REPORT TYPE		3. DATES COVERED 00-00-2012 to 00-00-2012				
4. TITLE AND SUBTITLE				5a. CONTRACT	NUMBER			
Status of the UV C	ure Powder Coating	g Demonstration Pro	oject	5b. GRANT NUMBER				
				5c. PROGRAM ELEMENT NUMBER				
6. AUTHOR(S)				5d. PROJECT NUMBER				
				5e. TASK NUMBER				
				5f. WORK UNIT NUMBER				
	ZATION NAME(S) AND AD 1 Laboratory/RXSS	` '	ı	8. PERFORMING REPORT NUMB	GORGANIZATION ER			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)					10. SPONSOR/MONITOR'S ACRONYM(S)			
			11. SPONSOR/MONITOR'S REPORT NUMBER(S)					
12. DISTRIBUTION/AVAIL Approved for publ	LABILITY STATEMENT ic release; distributi	on unlimited						
	SETSDefense 2012: -30, 2012, San Diego	-	inable Surface E	ngineering fo	or Aerospace and			
14. ABSTRACT								
15. SUBJECT TERMS								
16. SECURITY CLASSIFICATION OF: 17			17. LIMITATION OF	18. NUMBER	19a. NAME OF			
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified	Same as Report (SAR)	OF PAGES 69	RESPONSIBLE PERSON			

Report Documentation Page

Form Approved OMB No. 0704-0188

Outline

- Project Team
- UV Cure Technology
- UV Curable Powder Overview
- UV Cure Powder Coating
 Demonstration/Validation



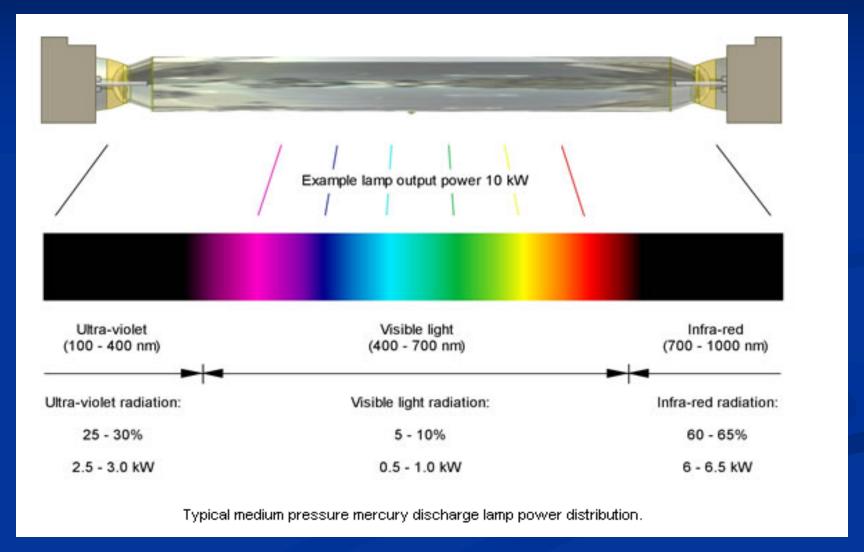


Project Team

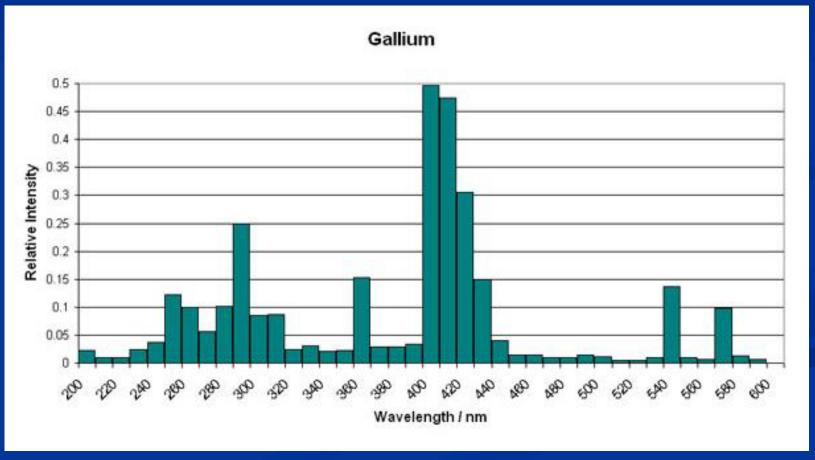
- Mr. William Hoogsteden, Principal Investigator Air Force Research Laboratory/RXSSO Wright-Patterson AFB, OH 45433 William.Hoogsteden@wpafb.af.mil (937) 656-4223
- Mr. Christopher W. Geib, Co-Principal Investigator Science Applications International Corp. 3745 Pentagon Blvd Beavercreek, OH 45431 Christopher.W.Geib@saic.com (937) 431-4332

Requires a source of UV light

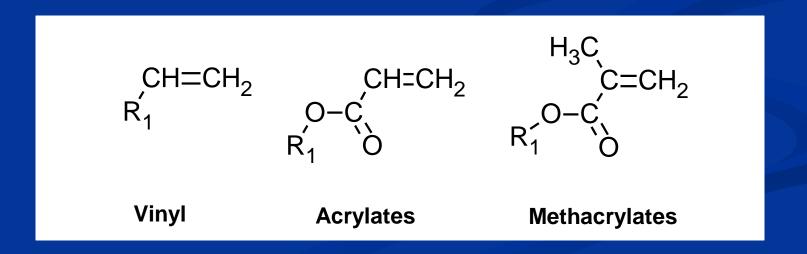




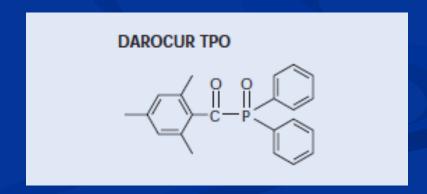
We use a Gallium doped lamp:



- Chemistry of UV-cure coatings
 - Can be virtually any polymer matrix used for organic coatings
 - The common denominator is the presence of a UV light reactive species on/in the polymer matrix
 - Commonly vinyl, acrylate or methacrylate groups



- UV Cure formulations require:
 - Light reactive polymer resins
 - Additives such as pigments and flow agents
 - Photoinitiators



- UV-cure powder coatings
 - Typically, the most common UV curable powders are:
 - Polyurethanes
 - Polyesters
 - Epoxies
 - Hybrids and mixtures of the above
 - For the UVCPC project, we use a special composition of light activated polyurethanes and polyesters

- Previous ways of thinking about powder
 - Coating cure temperatures typically above 220°C
 - Prohibitive for use on tempered metals (Al, Mg, Ti)
 - Prohibitive to use on composites
 - Powder coatings were designed as barrier protection

- Modern powder coatings can be formulated to have:
 - Lower melt & flow temperatures (< 110°C)
 - UV or EB cure functionality can be added
 - Various advanced nonchrome corrosion inhibitors



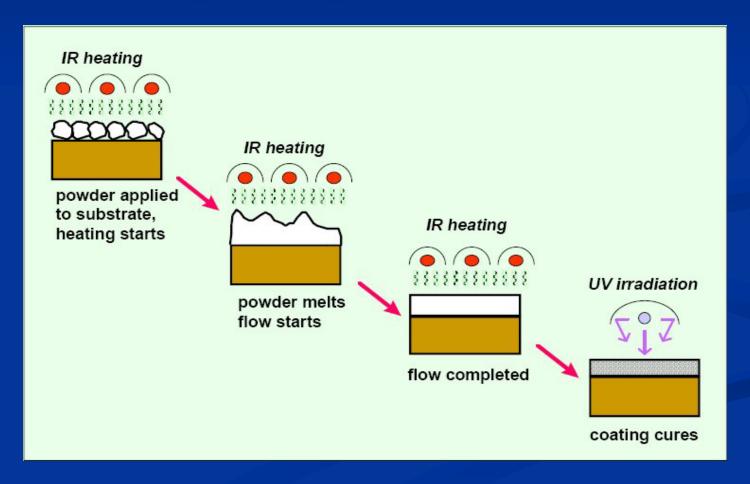
- Advantages of UV-cure powder coating:
 - Elimination of volatile organics (VOC)
 - Elimination of hazardous air pollutants (HAP)
 - Reduction/elimination of hazardous waste
 - Transfer efficiencies as high as 95% (w/reclaim)
 - Decrease in thermal exposure.
 - Large bulky parts that cannot fit into existing ovens can be coated and cured.
 - UV-cure powder requires less energy because the energy is focused to a specific part only as long as needed.



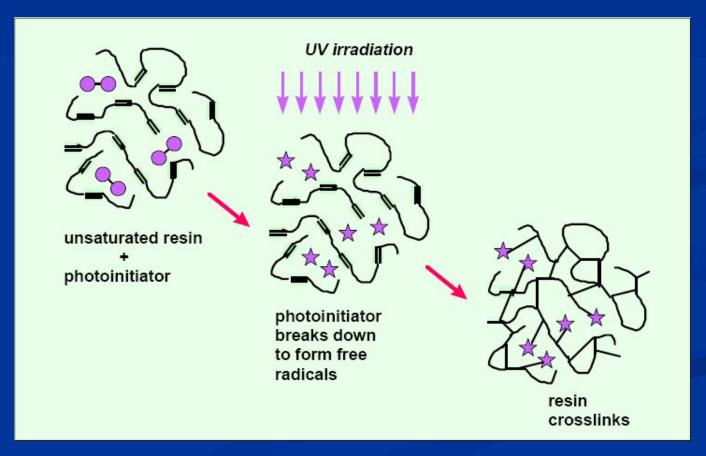


- Powder is applied using electrostatic powder gun
- Applied powder is cured with IR and UV lights mounted on robotic curing system

■ The UV cure powder process:



Crosslinking occurs during UV irradiation:



UV Cure Powder Coating Demonstration/Validation

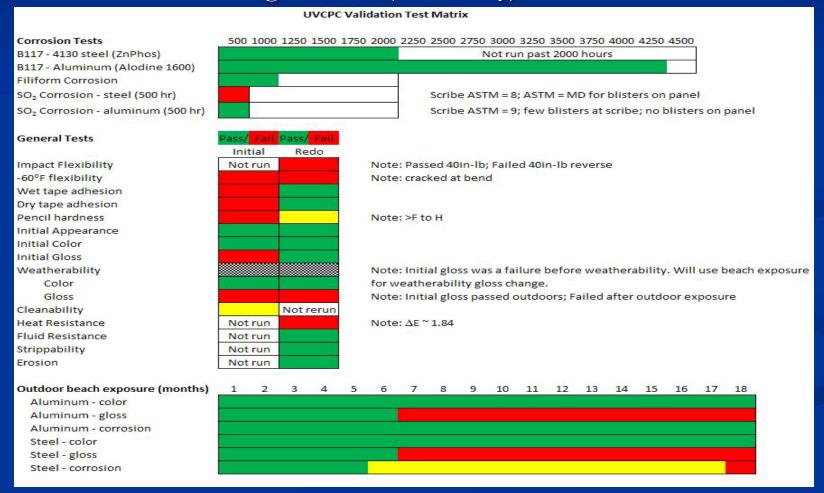
Timeline

- Project based on Commercial Off The Shelf (COTS) UV cured powder coatings
- Project started in 2008
- Initially had two powder vendors
- One dropped because of constant merger issues
- Initial validation testing completed in 2010
 - Results questionable due to adhesion issues
 - A number of tests rerun as a result
- Adhesion study completed in 2010
 - Found one of the reasons for poor adhesion

- Timeline (Cont.)
 - Adhesion study completed in 2010 (Cont.)
 - Low copper alloys (6000, 3000 series) not a problem
 - High copper alloys scavenge free radicals at surface
 - Determined that certain surface treatments are effective:
 - Anodized
 - Alodine 1600
 - Zinc Phosphate
 - Epoxy wash primers
 - Building 2801 modification completed end 2010
 - Robot installation occurred in 2011

- Timeline (Cont.)
 - First light and testing in early 2012
 - Discovery that kinetics also play major role in adhesion
 - First parts coated with UVCPC
 - Ammo can
 - Aircraft jack hydraulic reservoirs
 - USAF aircraft wheels

Validation Testing Results (Summary) of COTS UVCPC



- General test results
 - Color (FED-STD-595C)

Coating	L*	a*	b*	ΔL*	∆a*	Δb*	ΔE*
FED-STD-595C 17925 Reference Chip	96.06	-1.95	3.10				
PCRG High Gloss White	95.82	-1.96	2.66	-0.24	-0.01	-0.45	0.5
FED-STD-595C 26173 Reference Chip	55.05	-1.24	-3.66				
PCRG Semigloss Initial	55.13	-1.24	-3.98	0.08	0.00	0.32	0.2

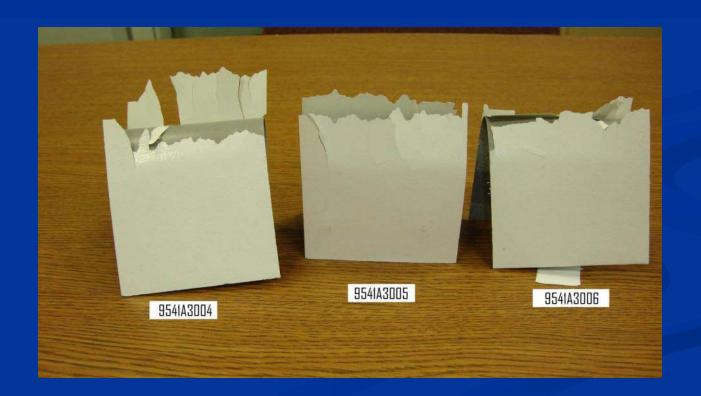
■ Gloss (FED-STD-595C)

Sample ID	20°	60°	85°
PCRG High Gloss White	55.1	84.4	95.6
PCRG Semi Gloss Initial	8.8	45.8	78.1

- General test results
 - Pencil Hardness (ASTM D3363)
 - Marginal, falls between F and H pencil
 - Impact Flexibility (MIL-PRF-85285D)
 - Passed 40 in-lb forward, Failed 40 in-lb reverse



- General test results
 - Low temperature (-60°F) flexibility initial (MIL-PRF-85285D)



- General test results
 - Low temperature (-60°F) flexibility rerun



- General test results
 - Dry/Wet tape adhesion (ASTM D3359, FED-STD-141D)
 - Initial results were failures due to adhesion issue
 - Dry adhesion was rerun on various pretreatments
 - Because adhesion seemed to change with time, a month of testing run
 - Summary of the dry tape adhesion results is shown on next slide

General test results

	Film Thickness	Cross Hatch Adhesion		931			×	X	
		1/5/2011	1/7/2011	1/10/2011	1/12/2011	1/14/2011	1/17/2011	1/19/2011	2/3/2011
Alodine 1200S									
45 sec	1.3 - 1.4	4B	4B	4B	48	48	2B	2B	N/A
90 sec	1.5 - 1.8	48	3B	3B	3B	3B	3B	2B	N/A
3 min	1.7 - 2.2	4B	.28	28	08	OB	OB	08	N/A
Alodine 1600									14111
1 min	1.7 2.1	5B	5B	5B	58	5B	5B	5B	4-5B
3 min	1.5 - 1.7	5B	5B	5B	5B	5B	4-5B	4-5B	4-5B
5 min	1.4 - 1.7	5B	5B	5B	58	5B	4-5B	4-5B	4-5B
20 sec	1.6 - 2.0	5B	5B	4-5B	4-5B	4-5B	4-5B	4-5B	4-5B
Alodine 5200									
1 min	1.3 - 1.5	5B	5B	5B	5B	5B	5B	5B	N/A
2 min	1.5 - 1.8	5B	5B	5B	5B	5B	5B	.5B	N/A
4 min	1.5 - 2.0	48	4B	4B	5B	4-5B	5B	5B	N/A
Alodine 5900									
5 min	1.3 - 1.4	4B	4B	3B	3B	28	08	OB	N/A
10 min	1.2 - 1.5	4B	3B	3B	28	28	OB	OB	N/A
Alodine 8800						American State of the Control of the			Laborate S
Heavy	1.6 -1.7	5B	5B	5B	58	5B	5B	5B	N/A
Light	1.5 - 1.7	5B	5B	5B	5B	5B	58	5B	N/A
Control	1.5 - 1.9	08	08	OB	08	08	08	08	N/A
Carpenter B/700		1.00						5B	33 days
S-W Wash Primer	Test	Initial + 2 week			·	·		·	
2024-T3	Dry	5B + no change							
2024-T3	Wet	5B + no change							

Dry

Wet

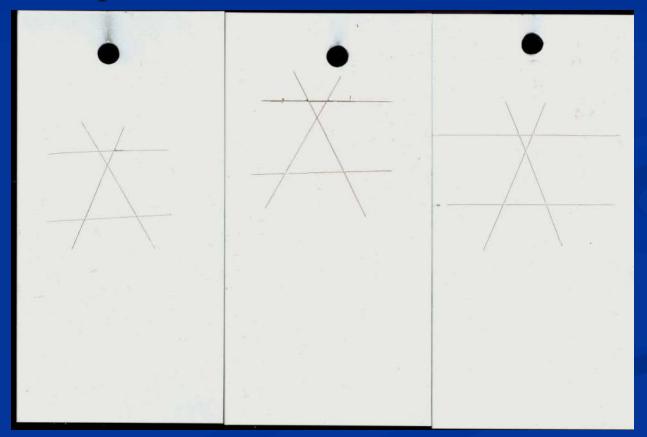
4130 steel

4130 steel

5B + no change

5B + no change

- General test results
 - Wet tape adhesion



- General test results
 - Fluid resistance (MIL-PRF-85285D)
 - Initial fluid resistance test halted as soon as adhesion issue discovered
 - Follow on fluid resistance test rerun passed



- General test results
 - Weatherometer (MIL-PRF-85285D, ASTM G155)
 - 500 hour test
 - $\Delta E^* = 0.97$ (Pass)
 - Gloss loss \approx 63.7 units (Fail)
 - Heat Resistance (MIL-PRF-85285D)
 - $\Delta E^* = 1.84$ (Marginal)
 - Cleanability (MIL-PRF-85285D)
 - Efficiency = 67% (Marginal)
 - Strippability (MIL-PRF-85285D)
 - 100% removed in < 4 hours (Pass)

- Corrosion resistance tests
 - Neutral salt fog (MIL-PRF-23377J, ASTM B117)
 - UVCPC over Zn Phosphate 4130 steel, 2000 hrs (Pass)
 - UVCPC over Alodine 1600, 2024-T3 Al, 4430 hrs (Pass)



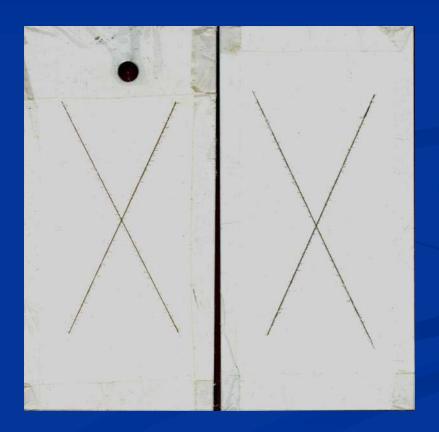


- Corrosion resistance tests
 - SO₂ corrosion resistance (ASTM G85, Annex 4)
 - UVCPC over Aluminum (Pass)
 - UVCPC over cold rolled steel (Fail)





- Corrosion resistance tests
 - Filiform corrosion resistance (MIL-PRF-23377J, ASTM D2803)
 - 1000 hour test (Pass)



- Erosion/Abrasion tests
 - Falling sand erosion testing (ASTM D968)
 - Within 1σ of the legacy coating

Falling Sand Evaluation (UVCPC)

Sample #	Liters (V)	Mean thickness (t)		A Factor A=V/t
2	144	2.32		62.1
3	162	2.8		57.9
4	144	2.53		56.9
5	133	2.53		52.6
6	144	2.58		55.8
8	143	2.49		57.4
			Mean	57.0
			Std Dev	3.09

- Long term outdoor exposure (ASTM D1014)
 - Three parameters evaluated
 - Color drift
 - Gloss drift
 - Overall corrosion
 - Semi-gloss gray UVCPC used
 - Results:
 - Color drift maximum $\Delta E^* = 0.82$ (Pass)
 - Gloss drift 36.6 gloss units (Fail)
 - Corrosion overall:
 - Aluminum still passing after 18 months (~12900 hours exposure)
 - Cold rolled steel failed after 7 months (~5000 hours exposure)

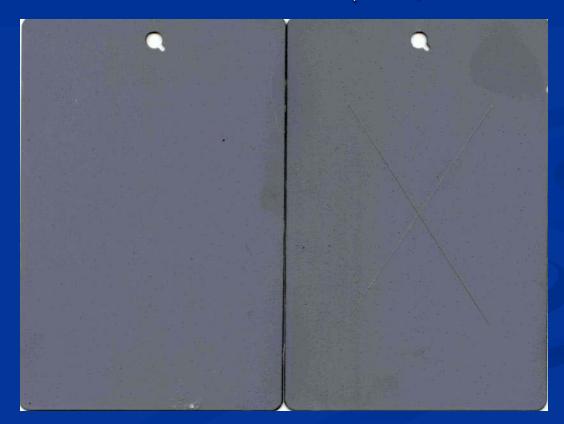
- Long term outdoor exposure
 - Color drift

Coating	L*	a*	b*	ΔL^*	Δa*	Δb*	Δ E*
FED-STD-595C 26173 Reference Chip	55.05	-1.24	-3.66				
PCRG Semigloss Initial	55.13	-1.24	-3.98	0.08	0.00	0.32	0.2
PCRG 7-month color	55.40	-1.18	-4.16	0.35	-0.06	0.50	0.32
PCRG 12-month color	55.77	-1.16	-4.11	0.72	-0.08	0.45	0.75
PCRG 18-month color	55.82	-1.21	-4.17	0.76	-0.07	0.31	0.82

- Long term outdoor exposure
 - Gloss drift

Sample ID	20°	60°	85°
PCRG Semi Gloss Initial	8.8	45.8	78.1
PCRG 7-month semigloss	3.0	25.7	66.9
PCRG 12-month semigloss	2.2	21.9	60.2
PCRG 18-month semigloss	0.9	9.2	41.6

- Long term outdoor exposure
 - Aluminum after 18 months (~12,900 hours)



- Long term outdoor exposure
 - Steel after 7 & 18 months





- Actual components coated
 - Ammunition can



- Actual components coated
 - Aircraft jack hydraulic reservoirs





Actual components coated

■ F-16 main wheel (before)









- Actual components coated
 - F-16 main wheel (after)





- Actual components coated
 - F-15 nose wheel (before)





- Actual components coated
 - F-15 nose wheel (after)



- Actual components coated
 - Coast Guard MC-130 landing gear door



Summary

- Overall, the COTS UVCPC did well
 - Better overall test results than previous Low Temp powder
 - Positives
 - Excellent B117 corrosion resistance over aluminum
 - Good corrosion resistance over zinc phosphated steel
 - Excellent Filiform corrosion resistance
 - Good room temperature flexibility
 - Erosion resistance on par with legacy 2K coatings

Summary

- Overall, the COTS UVCPC did well (cont.)
 - Could use some improvements going forward
 - Coating
 - Lower melt/flow temperature
 - Improve -60°F flexibility
 - Increase hardness to 2H or greater pencil
 - Improve impact flexibility
 - Better heat resistance
 - Improve weatherability (gloss)
 - Reformulate for direct-to-metal

Summary

- Overall, the COTS UVCPC did well (cont.)
 - Could use some improvements going forward
 - Robotics
 - Better profiling
 - Use profiling radiometers
 - Better thermal profiling
 - Better control during operations (thermal, UV)
 - IR and UV feedback to robot
 - Powder Coating
 - Incorporate non-contact uncured powder thickness gauge

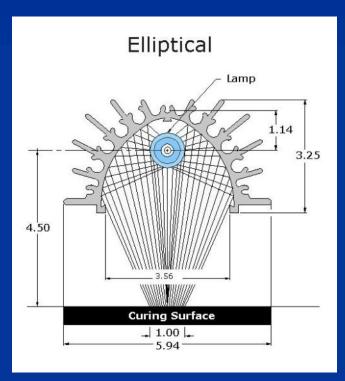
Questions?

UVCPC Back up slides

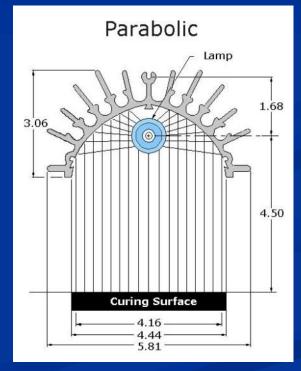
- Adhesion of UVCPC over 2000 series aluminum
 - Adhesion results could not be duplicated between CTIO and PCRG
 - Key differences between locations was power of UV lamps
 - Formulation developed under a 300 Watt/in lamp
 - Originally thought it was photoinitiator based
 - "Flash" effect considered
 - Determined to test on the robotic curing system at NASWI
 - Nordson lamp is power adjustable unlike the CTIO/PCRG lamps
 - Robot can duplicate conveyor speeds (5 fpm vs. 9 fpm)
 - Robot can execute multiple passes in programming
 - Felt that the system could duplicate either lab
 - However, the results were completely unexpected
 - Realized the lamp at NASWI is a non-focused lamp
 - NASWI results led to the belief that both chemistry and kinetics play a role in the cure and adhesion on metallic substrates

- Adhesion of UVCPC over 2000 series aluminum
 - The robotic curing system was able to cure with 5B adhesion
 - 5086, 6061, 3003 aluminum, and 4130 steel
 - None of the test panels had been prepared
 - Wiped free of dust
 - No pretreatment
 - No scuffing of surface (except steel which was bead blasted)
 - On 2000 series untreated, unprepared aluminum, 3B to 4B adhesion was possible
 - Kinetics plays a role as well as free radical scavengers
 - Free radical concentration at an instant in time
 - Focused lamps vs. unfocused lamps

- Adhesion of UVCPC over 2000 series aluminum
 - Focused vs. unfocused reflectors



Used by CTIO and PCRG

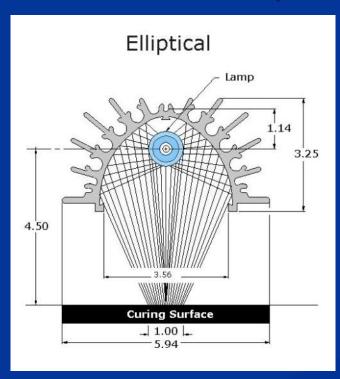


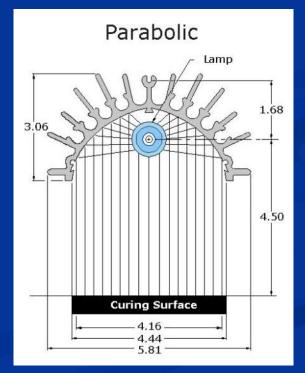
Used by NASWI

- Adhesion of UVCPC over 2000 series aluminum
 - Focused vs. unfocused reflectors
 - Dose at each location (typical)

	WPAFB lamp (focused) J/cm ² 2-pass, 9 fpm	PCRG lamp (focused) J/cm ² 2-pass, 5 fpm	NASWI lamp (unfocused) J/cm ² 1-pass, ~4 fpm
UVA	2.54	2.22	1.53
UVB	1.83	1.39	1.62
UVC	0.13	0.17	0.22
UVV	12.7	10.41	12.23

- Adhesion of UVCPC over 2000 series aluminum
 - Focused puts almost full dose in a 1" path
 - Unfocused puts similar dose down across ~4" path
 - Between 0.6 (WPAFB) and 1 (PCRG) second for full dose in focused
 - About 5 seconds (NASWI) for full dose in unfocused





- Adhesion of UVCPC over 2000 series aluminum
 - Kinetics of the cross linking reaction in UVCPC
 - **■** Time based equations

$$v_i = 2k_d f[I]$$

Eq. (1) initiation

$$v_p = k_p[M][M \cdot]$$

Eq. (2) propagation

$$v_t = 2k_t[M \cdot]^2$$

Eq. (3) termination

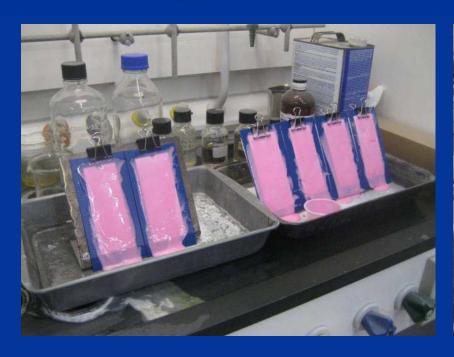
- Adhesion of UVCPC over 2000 series aluminum
 - Concentration of free radicals directly related to dose received
 - For a given "instant" in time:
 - WPAFB instantaneous free radical conc. is 3.4 times PCRG lamp
 - WPAFB instantaneous free radical conc. is 5.4 times NASWI lamp
 - Results in a relative increase of 11.6, or 29 time increase in v_t between WPAFB, PCRG, and NASWI lamps

$$v_p = k_p[M][M \cdot]$$
$$v_t = 2k_t[M \cdot]^2$$

- If v_t is $\geq v_p$, then:
 - Premature termination
 - Excessive shrinkage
 - Low cross link density
 - No or poor adhesion

- Summary of Adhesion Issue:
 - Copper or other free radical scavengers have an effect
 - Scavenger "effect" can be overcome with:
 - Certain chromate conversion coatings
 - Anodizing
 - Epoxy based wash primer
 - Adjustment "tweak" in formulation
 - Kinetics based on free radical concentration at an instant in time
 - Overcome termination rate by spreading the dose
 - A little longer cure is a "better" cure (5 seconds vs. 1 second)
 - These factors have now been demonstrated by actual test

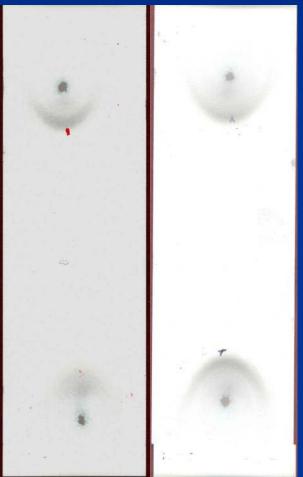
- General test results
 - Strippability



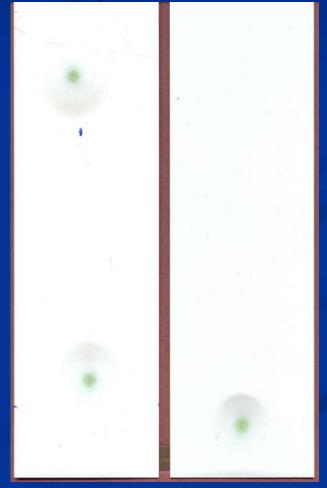


Falling sand testing

UVCPC over 2024 Anodized



Legacy over 2024 Anodized



- Actual components coated
 - Coast Guard MC-130 landing gear door
 - Entrained moisture created huge bubble during IR phase





- Actual components coated
 - Coast Guard MC-130 landing gear door
 - Entrained moisture created huge bubble during IR phase





- Actual components coated
 - Coast Guard MC-130 landing gear door
 - Entrained moisture created huge bubble during IR phase



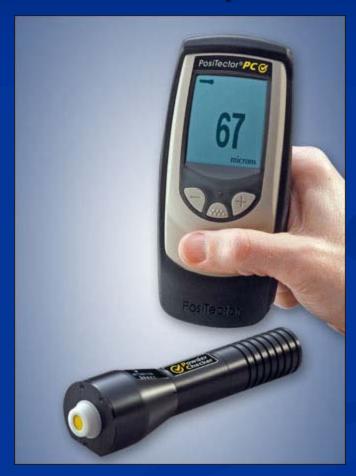


- Better Robotic Profiling
 - Use of small radiometers
 - Extended use of thermal profiling





- Better Powder Coating
 - Use of non-contact uncured powder thickness gauge



- Estimated Cost of Improvements
 - Hardware \$6K
 - Robotics modifications \$12K
 - Coatings reformulation and revalidation \$120K